

Short communication

Removing polysaccharides-and saccharides-related coloring impurities in alkyl polyglycosides by bleaching with the H_2O_2 /TAED/ NaHCO_3 system

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ABSTRACT

The effect of H_2O_2 /TAED/ NaHCO_3 system, namely NaHCO_3 as alkaline agent with the (tetra acetyl ethylene diamine (TAED)) TAED-activated peroxide system, bleaching of alkyl polyglycosides solution was studied by spectrophotometry. The results showed that the optimal bleaching conditions about H_2O_2 /TAED/ NaHCO_3 system bleaching of alkyl polyglycosides solution were as follows: molar ratio of TAED to H_2O_2 was 0.06, addition of H_2O_2 was 8.6%, addition of NaHCO_3 was 3.2%, bleaching temperature of 50–65 °C, addition of MgO was 0.13%, and bleaching time was 8 h. If too much amount of NaHCO_3 was added to the system and maintained alkaline pH, the bleaching effect would be greatly reduced. Fixing molar ratio of TAED to H_2O_2 and increasing the amount of H_2O_2 were beneficial to improve the whiteness of alkyl polyglycosides, but adding too much amount of H_2O_2 would reduce the transparency. In the TAED-activated peroxide system, NaHCO_3 as alkaline agent and buffer agent, could overcome the disadvantage of producing black precipitates when NaOH as alkaline agent.

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1. Introduction

Alkyl polyglycosides are products which are obtained under the catalysis of acid by losing a molecule of water from hemiacetal hydroxyl of glucose and fatty alcohol hydroxyl. The products are not a pure compound, but alkyl glycoside with different degree of glucose polymerization, commonly known as alkyl polyglycosides (abbreviation APGs) (Czichocki, Fiedler, Haage, Much, & Weidner, 2002; Wang, 2001). Alkyl polyglycosides are the fourth generation of world-class green surfactant developing in the 1990s which

do not use petroleum-derived products. APGs have many advantages of both nonionic and anionic surfactants. In addition to their good dermatological compatibility, biodegradability and excellent behavior at interfaces, APGs show interesting application properties, specifically for applications such as detergents, cleaning agents, cosmetic products and pesticide formulations (Hill, von Rybinski, & Stoll, 1997; Kahl, Quitzsch, & Stenby, 1997; Ruegsegger, Zhang, & Marchant, 1997; von Rybinski, 1996).

Generally pure alkyl polyglycosides are white powder (Wang, 2001). However, in practical industrial synthesis, recombination reaction and decomposition reaction are liable to occur at high temperature and acid environment, generating polysaccharides and other colored substances. The color bodies were presumed as polymerized dehydration and degradation products of saccharides, such as levulinic acid, furfural, or hydroxymethyl furfural. These polymers, known as humins, show conjugated unsaturated moieties that absorb light and may be responsible for the dark-hued

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discoloration of the alkyl polyglucosides (Balzer & Lüders, 2000). Alkyl polyglycosides are generally brown after dealcoholization reaction, which seriously affects its application scope and commercial value. Therefore, bleaching of alkyl polyglycosides solution becomes extremely important.

The well-known environmentally safe bleaching method is hydrogen peroxide bleaching (Hou, Zhang, & Zhou, 2010). While only using hydrogen peroxide, the bleaching effect is not satisfactory, so bleach activators need to be added. The most commonly used bleach activator is tetra acetyl ethylene diamine (TAED). It has been considered to be a non-toxic, non-sensitive and non-mutagenic product which can be degraded by microorganism to form carbon dioxide, water, ammonia and nitrate (Chen, 2008; Hsieh, Agrawal, Maurer, & Mathews, 2006). The main advantage of TAED-activated peroxide bleaching is that the reaction can be conducted at low temperature and near-neutral pH conditions (Xu, Long, Du, & Fu, 2013). In this paper the method of H_2O_2 /TAED/ NaHCO_3 system bleaching of alkyl polyglycosides was studied, and the effect of TAED, H_2O_2 , NaHCO_3 , MgO bleaching temperature and bleaching time on extinction coefficient was investigated.

2. Experimental

2.1. Materials

Alkyl polyglycosides (unbleached) was kindly provided by Shijiazhuang Jin Mo Er Chemicals Co., Ltd., China. TAED (purity 92%) was purchased from Zhejiang Jinke Chemicals Co. Ltd., China. Sodium bicarbonate was used as alkaline agent & buffer agent and purchased from Tianjin Yingda Rare Chemical Reagents Factory, China. Magnesium oxide was used as stabilizer and purchased from Tianjin Fengchuan Chemical Reagent Science And Technology Co., Ltd., China. Hydrogen peroxide (30%) & sodium hydroxide were purchased from Tianjin Hedong Hongyan Chemical Reagent Factory, China. All other chemicals were of analytical grade unless otherwise stated. Distilled water was used to experiment test.

2.2. Bleaching method

Unbleached alkyl polyglycosides were used as the experimental material in the bleaching process. The solid content of unbleached alkyl polyglycosides was 62.1%. In each bleaching experiment the dosage of alkyl polyglycosides was 50 g, and stirrer speed was 150 r/min. Firstly, the water bath was set at a certain temperature (45–70 °C), then MgO (0–0.12 g that was 0–0.39% of APGs, in this paper all the data were based on the solid content of APGs), NaHCO_3 (0.5–1.4 g that was 1.6–4.5% of APGs), TAED and H_2O_2 (5–10 ml, and the molar ratio of TAED to H_2O_2 was 0.05–0.07) were added in turn, in which H_2O_2 was added in batches to prevent the materials from overflowing. Additionally adding H_2O_2 in batches could make H_2O_2 and peracetic acid participate in bleaching reaction timely so as to reduce their ineffective decomposition. The concrete operations were adding 1–2 ml H_2O_2 each time, and adding once every hour. Under stirring, the whole bleaching reaction was maintained for 8 h (including H_2O_2 addition time and subsequent bleaching time). After that, the pH value (10% APGs solution) was adjusted to 11.5–12.5 with NaOH.

2.3. Analysis

The absorbance of alkyl polyglycosides solution after bleaching was measured at wave length of 470 nm with spectrophotometer (721G, Shanghai Precision and Scientific Instrument Co., Ltd., China). The color of solution was characterized by extinction



Scheme 1. H_2O_2 dissociation.

coefficient. The calculation formula is shown by Eq. (1) (Roth, Edwards, & Patrick McCurry, 2001):

$$E_{470} = \frac{A}{(C \times L)} \quad (1)$$

where E_{470} is extinction coefficient at 470 nm, A is measured absorbance of APGs solution at 470 nm, C is concentration in grams per cm^3 , and L is path length in centimeters.

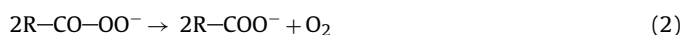
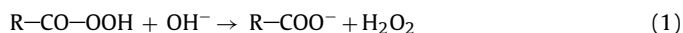
3. Results and discussion

3.1. Theoretical base of the TAED/ H_2O_2 / NaHCO_3 system

TAED-activated peroxide system under alkaline condition reacts as follows:

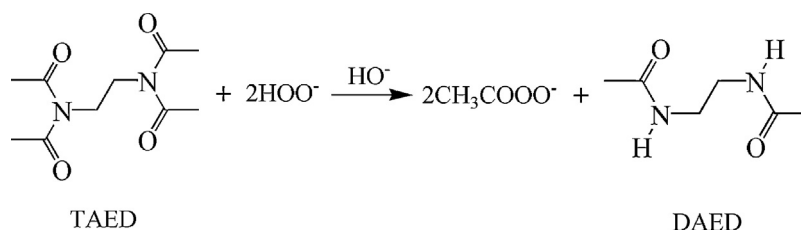
HOO^- is believed to be the effective bleaching species. Under alkaline condition, H^+ will be reduced, thus promoting the dissociation of H_2O_2 (as shown in Scheme 1). Perhydrolysis and hydrolysis of TAED will take place in alkaline solutions, as shown in Scheme 2 (Davies & Deary, 1991; Suchy & Argyropoulos, 2001) and Scheme 3 (Cai, Evans, & Smith, 2001; Davies & Deary, 1991; Scarborough & Mathews, 2000; Shao, Huang, Wang, & Liu, 2010). Because the nucleophilicity of perhydroxyl anions is far higher than the nucleophilicity of hydroxyl anions in aqueous solution, TAED reacts preferentially with perhydroxyl ion to release a stable peracid. It has been postulated that TAED forms hydrogen bonds with hydrogen peroxide during perhydrolysis, thus stabilizing the transition state and favoring perhydrolysis. Moreover, TAED does not form diacyl peroxides (Milne, 1998). Thus, perhydrolysis of TAED is the main reaction under alkaline condition. The oxidation potential of peracetic acid (1.81 eV) is higher than oxidation potential of hydrogen peroxide (1.33 eV) (Xu & Cha, 2012). So generally peracids are considered to be better and more active bleaching agents than peroxide (Leduc, Montillet, & Daneault, 1998).

However, under alkaline environment, generated peracids would be decomposed in the following ways:

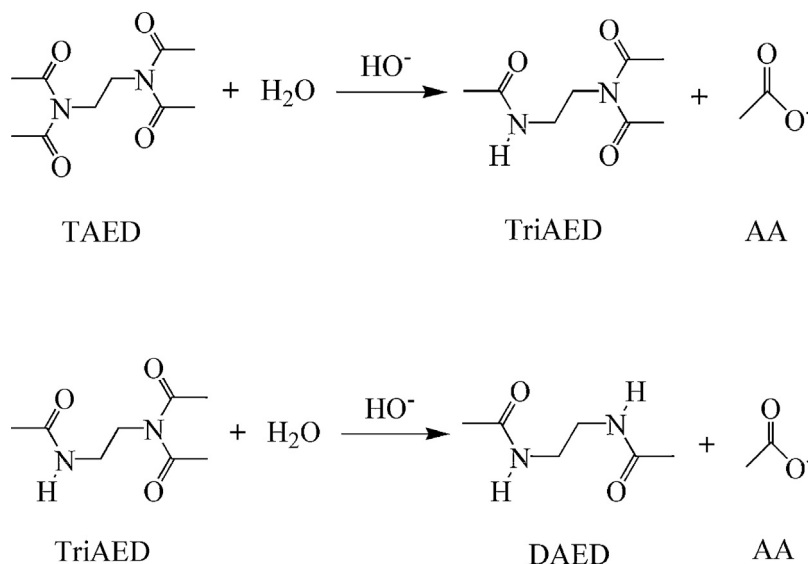


R is alkyl. In most cases, formula (2) is the main reaction (Deng & Feng, 2005; Reichert, McNeight, Elston, & Niagara Falls, 1944).

NaOH was usually used as alkaline agent in hydrogen peroxide bleaching of alkyl polyglycosides. While TAED-activated peroxide system bleaching of alkyl polyglycosides has higher requirements for the solution pH. Using NaOH as alkaline agent, the black precipitate could be formed easily in the APGs solution after bleaching, which affected the product quality. For the TAED-activated peroxide system, the active ingredient peracetic acid released acetic acid, and pH value decreased gradually with the process of bleaching reaction, which would lead to the perhydrolysis of TAED decelerated or eventually terminated. Hence a buffer agent was required to sustain the acid and alkali environment. In this study NaHCO_3 was to be thought of the appropriate alkaline agent. As strong base-weak acid salt, NaHCO_3 has strong ability to cushion and stabilize solution, meeting the requirements of system pH. More importantly, it basically avoided the disadvantage of producing black precipitate when NaOH as the alkaline agent.



Scheme 2. Perhydrolysis of TAED.



Scheme 3. Hydrolysis of TAED.

3.2. Effect of variables on bleaching performance

3.2.1. Effect of TAED

Fixing the amount of H_2O_2 and changing adding quantity of TAED, which can also be expressed by molar ratio of TAED to H_2O_2 , the relation between TAED dosage and extinction coefficient of APGs solution was shown in Fig. 1. It can be seen from the figure that the extinction coefficient was low (extinction coefficient was 0.204–0.201) when the amount of TAED was 0.70–0.75 g, and the corresponding molar ratio of TAED to H_2O_2 was 0.057–0.061, which

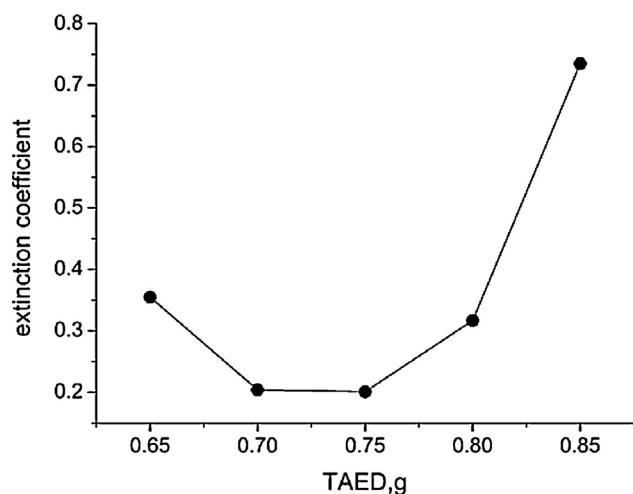


Fig. 1. The relation between TAED addition and extinction coefficient (note: bleaching was performed at 60 °C using 0.04 g MgO, 0.5 g NaHCO_3 and 5.5 ml H_2O_2).

differed considerably from 0.5, the theoretical mole ratio of the reaction between TAED and H_2O_2 (as shown in Schemes 1 and 2). Relative to the theoretical value, the experimental results showed H_2O_2 was overdosing. It was most likely because of the invalid decomposition of part of H_2O_2 during the reaction process; in alkaline condition, HOO^- was generated by the decomposition of H_2O_2 , and in addition to some reacting with TAED to produce peracetic acid (PAA) with high activity (Scheme 2), others were directly used for bleaching reaction; when the solution pH was adjusted to 11.5–12.5 by adding NaOH after bleaching, the whiteness of APGs improved again, indicating that there was an excess of H_2O_2 , which produced HOO^- to bleaching again. These reasons caused the adding amount of H_2O_2 much larger than theoretical amount of the reaction between TAED and H_2O_2 . As shown in Fig. 1, when TAED was 0.75 g corresponding the molar ratio of TAED to H_2O_2 approximately 0.06, the bleaching effect was the best.

3.2.2. Effect of H_2O_2

Fig. 2 showed the extinction coefficient of alkyl polyglycosides solution bleached using H_2O_2 with and without the addition of TAED. It could be seen that the extinction coefficient of solution decreased obviously and the color of solution was significantly better by adding TAED than only H_2O_2 . So TAED-activated peroxide system bleaching of alkyl polyglycosides solution could effectively improve the solution color. When adding TAED, the extinction coefficient was reduced with the increase of H_2O_2 amount, and the solution color became lighter. When the amount of H_2O_2 continued to increase ($\text{H}_2\text{O}_2 > 8$ ml), the extinction coefficient was slightly increased, and at the same time it could be found through the observation of the sample solution that the solution was light in color but the transparency decreased. While the best bleaching effect was reflected by both light color and high transparency. Thus it could

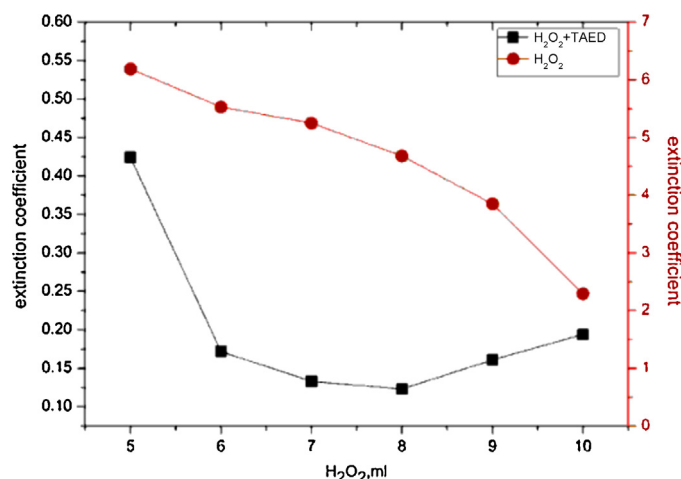


Fig. 2. The relation between H₂O₂ addition and extinction coefficient (note: bleaching was performed at 60 °C using 0.04 g MgO, 0.5 g NaHCO₃ and the molar ratio of TAED to H₂O₂ was 0.061).

be seen from Fig. 2 that when H₂O₂ addition was 8 ml that was 8.6% of APGs, the best bleaching effect was obtained.

3.2.3. Effect of NaHCO₃

As shown in Fig. 3, the extinction coefficient of the solution decreased gently with the increase of NaHCO₃ in the initial stage, and the solution color is light. When NaHCO₃ addition amount was greater than 1.0 g, the extinction coefficient increased obviously, and the solution color became darker significantly. It could be seen from Fig. 4 that with the increase of NaHCO₃ before bleaching, there was no obvious differences among the pH value, basically stable at around 10.5. After bleaching the pH of the first three groups have experienced the transition from base to acid, while the latter two groups were still maintained in an alkaline environment before and after bleaching. In alkaline environment in addition to a small amount of TAED was used in hydrolysis reaction most of them was used in perhydrolysis reaction to generate large amounts of peracetic acid (shown in Schemes 2 and 3, respectively). While the sustained alkaline environment made peracetic acid cannot react effectively with the chromophore and decomposed ineffectively (shown in formula (1) and (2)), which made the bleaching effect worse. The first three groups meet the demand of the pH value of the system. At the beginning of the reaction peracetic acid was

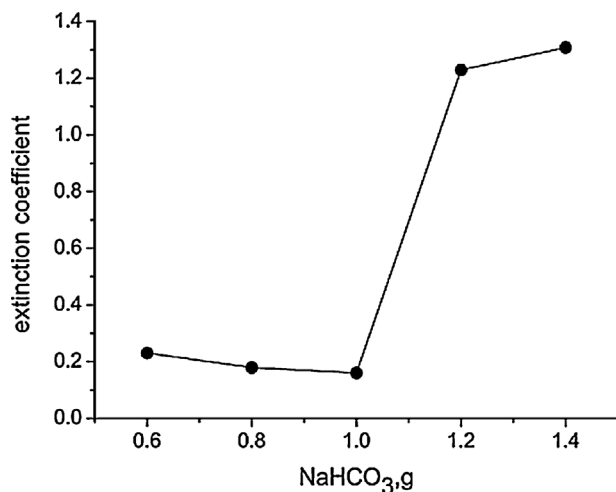


Fig. 3. The relation between NaHCO₃ addition and extinction coefficient (note: bleaching was performed at 50 °C using 0.1 g MgO, 1.064 g TAED and 9 ml H₂O₂).

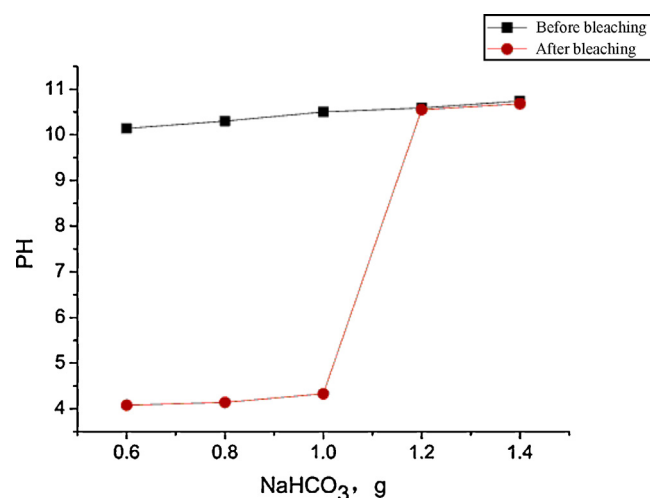


Fig. 4. Changes of PH before and after bleaching.

produced in the system, while as the bleaching progressed peracetic acid decomposed to produce acetic acid, resulting that the solution pH reduced to neutrality or acidity. At the moment peracetic acid could effectively play the role of bleaching, to improve the bleaching effect. Thus, adding NaHCO₃ could adjust the pH value of the system, and then affected the bleaching effect of APGs. It could be seen from Fig. 3, the addition amount of NaHCO₃ for the extinction coefficient or bleaching effect existed a turning point (1 g). When the dosage of NaHCO₃ was lower than the turning point, the bleaching effect was better. But when it exceeded the turning point, the bleaching effect was significantly worse. At the turning point, the extinction coefficient was lowest and the solution color was the lightest. Therefore, the optimum addition level of NaHCO₃ was 1 g that was 3.2% of APGs.

3.2.4. Effect of temperature

The reaction rate increased as the temperature rose. For peracetic acid, the higher the temperature, the faster the decomposition rate, the worse the stability (Li, 1984). The most effective way of bleaching was the formation rate of the effective bleaching agents (PAA and HOO⁻) was nearly equal to the reaction rate between bleaching agents and chromophores. If the temperature was too high, the reaction rate increased, and the decomposition was accelerated, which would make parts of bleaching components be decomposed before they react with the chromophores. If the temperature was too low, the formation rate of effective bleaching ingredients would slow down, influencing the bleaching effect within the same time. It could be seen from Fig. 5 that in the initial phase, the extinction coefficient decreased gradually with the increase of temperature, the solution color became light. But the color would become darker when the temperature was too high (such as 70 °C). Therefore the preferred temperature range was 50–65 °C.

3.2.5. Effect of MgO

Mg²⁺ was a hydrogen peroxide stabilizer, because magnesium salt reacts with alkali and generates Mg(OH)₂ which could form peroxy chain with hydrogen peroxide, making hydrogen peroxide stable and preventing its violent decomposition, to improve the bleaching effect of hydrogen peroxide (Wang, 2001). Meanwhile, it was found from the polarographic study that magnesium salt did not react with H₂O₂ but with the metal ion catalyst, thus affecting its activity and playing a controlling role in the decomposition of H₂O₂ (Geng, Zhang, Dou, Zheng, & Qin, 1996). As shown in Fig. 6, without the addition of MgO the extinction coefficient was high

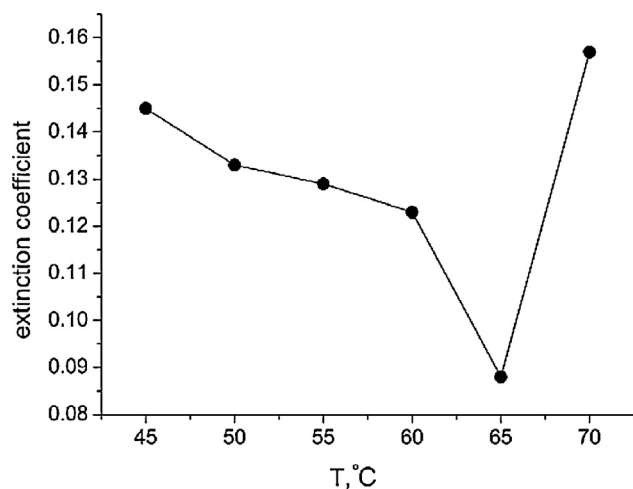


Fig. 5. The relation between temperature and extinction coefficient (note: bleaching was performed using 0.04 g MgO, 0.5 g NaHCO₃, 1.091 g TAED and 8 ml H₂O₂).

and the bleaching effect was poor. While the extinction coefficient significantly reduced by adding a small amount of MgO. When MgO continued to increase, the extinction coefficient is slightly higher and bleaching effect starts to deteriorate. This illustrated that the MgO stabilized H₂O₂, and simultaneously self-generated Mg(OH)₂ precipitation under alkaline environment would affect the appearance of the product. It could be seen from Fig. 6 that when the dosage of MgO was 0.04 g that was 0.13% of APGs, the optimum bleaching effect could be obtained.

3.2.6. Effect of time

In the experiment 8 ml H₂O₂ was added in batches in which 2 ml H₂O₂ was added once every hour. As is known, alkyl polyglycosides are surfactants, which have high foaming ability. At higher temperatures, the APGs solution would easily overflow if too much H₂O₂ was added at a time. Additionally adding H₂O₂ in batches could make H₂O₂ and peracetic acid participate in bleaching reaction timely, so as to reduce the ineffective decomposition. After the addition of 8 ml H₂O₂ in the first 3 h, sampling and testing started once every hour from the 4th hour viz. the completion of adding H₂O₂ for the accuracy of the experiment.

The bleaching time included the main bleaching time and subsequent bleaching time. The first 4 h were the main bleaching time. In this process H₂O₂ was added once every hour, a large amount of

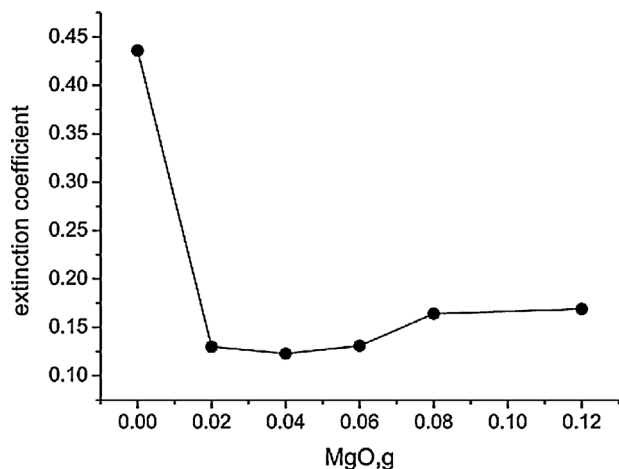


Fig. 6. The relation between MgO addition and extinction coefficient (note: bleaching was performed at 60 °C using 0.5 g NaHCO₃, 1.091 g TAED and 8 ml H₂O₂).

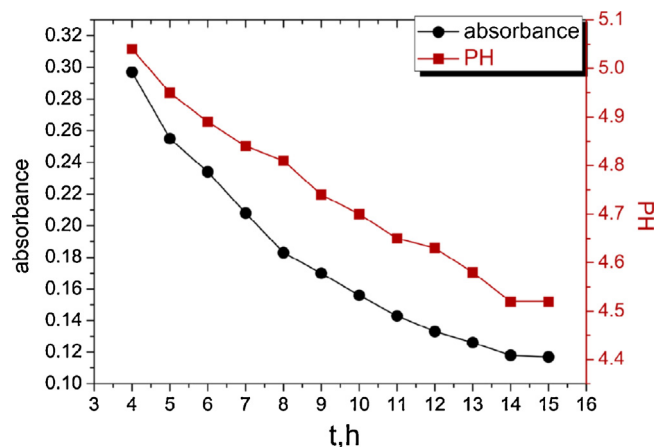


Fig. 7. Effect of subsequent bleaching time on absorbance and PH (note: bleaching was performed at 60 °C using 0.02 g MgO, 0.5 g NaHCO₃, 1.091 g TAED and 8 ml H₂O₂).

peracetic acid was produced continuously in the system and played the role in bleaching performance. At this stage the color of the alkyl polyglycosides solution changed significantly from dark brown to light yellow. Four hours later the addition of H₂O₂ completed (the solution was acidity), the generation rate of peracetic acid became slow, but within a period of time the bleaching reaction was still in progress, and the color became lighter. This period of time was the subsequent bleaching time, the results were shown in Fig. 7.

In this paper, the color of solution was characterized by extinction coefficient. Eq. (1) $E_{470} = A/(C \times L)$, in which the path length (L) is 1 cm. When all H₂O₂ has been added, the solution mass concentration (C) basically unchanged. So the changes of the extinction coefficient (E_{470}) depended on the absorbance (A), that is the variation tendency of the solution color could be characterized by the variation tendency of the absorbance. In the experiment the samples were diluted 2-fold to detect absorbance and 10-fold to detect pH. As seen in Fig. 7, with the increase of bleaching time, the pH value and the absorbance both decreased. Before 8 h the degrade gradient of the absorbance was larger, while 8 h later the degrade gradient of the absorbance was relatively small. At 4 h, 8 h and 14 h, the absorbance value of the solution was 0.297, 0.183 and 0.118, respectively. From 4 h to 8 h the absorbance of the solution dropped 0.114, while from 8 h to 14 h the absorbance of the solution dropped only 0.065. Comprehensive consideration of various factors, the optimum bleaching time was about 8 h.

4. Conclusions

TAED as the bleach activator is often applied in bleaching of pulp, fiber, fabric, etc. While this paper emphasized on the study of TAED application in bleaching of alkyl polyglycosides. Besides, in the TAED-activated peroxide system, NaHCO₃ was chosen as alkaline agent and buffer agent, which has overcome the disadvantage of producing black precipitates when NaOH as alkaline agent. If too much amount of NaHCO₃ was added to the APGs solution and maintained alkaline pH, the bleaching effect would be greatly reduced. When the molar ratio of TAED to H₂O₂ was fixed, increasing the amount of H₂O₂ could improve the whiteness of APGs, but adding too much amount of H₂O₂ would reduce the transparency of APGs solution. Research results showed that the best bleaching conditions about H₂O₂/TAED/NaHCO₃ system bleaching of alkyl polyglycosides solution were as follows: molar ratio of TAED to H₂O₂ was 0.06, addition of H₂O₂ was 8.6%, addition of NaHCO₃ was 3.2%, bleaching temperature was 50–65 °C, addition of MgO was 0.13%, and bleaching time was 8 h.

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